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PARTIAL DEMINERALIZATION OF SALINE WASTES

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PARTIAL DEMINERALIZATION OF SALINE SEWAGE BY ION EXCHANGE

PROGRESS REPORT NO. 3

PARTIAL DEMINERALIZATION OF SALINE WASTES

By

R. L. Sands and W. J. Kaufman

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RECYCLING OF SALINE WASTES

by

R. L. Banks and W. J. Kaufman

INTRODUCTION

The continually increasing demands for fresh water, combined with dwindling reserves, have prompted an extensive effort to find economical processes for the production of drinking water from sea water, brackish water, and waste water. The recycling of waste water may offer the advantages of close proximity of source to demand, comparatively low mineral content, and the potential for alleviating pollution in the same operation. Attention has been focused upon the utilization of waste water for ground water recharge by surface spreading, by injection into wells, and by spraying onto forests or croplands. Utilization by industry has also been considered and practiced and the use of sewage for irrigation is a well established though not necessarily commendable practice. Since communities commonly obtain their water supply from rivers receiving the wastes of upstream neighbors, the partial recycling of wastes is in a sense already widespread and there has been at least one instance of the deliberate and direct recycling of sewage to meet emergency municipal needs [1].

With each use some impairment of quality occurs due to the addition of living organisms and organic and mineral substances, all of which may be termed the "use increment." Representative values of the use increment as measured by several indices are given in Table I. It is evident that the increment is quite variable and that it may depend on the

conditions of secondary treatment as well as on the particular uses of water in the community. A major effort has been made to discover and evaluate processes for the removal of the use increment by the Advanced Waste Treatment Research program [2] conducted by the Robert A. Taft Sanitary Engineering Center. The most promising of the processes studied in this program were adsorption, foam separation, distillation, and electrodialysis. In the authors' opinion, other processes also showing promise include reverse osmosis, which provides ultra-filtration as well as desalination, sewage spreading and ground-water recharge, which reduces nitrogen and phosphorus; algae ponds, which are effective in reducing BOD, COD, nitrogen, phosphorus, ammonia, and hardness; and ion exchange. Ion exchange has a number of advantages over other processes: (1) undesirable ions can often be selectively removed, (2) its controllability permits a wide range of effluent quality, (3) there is an established technology, (4) it is well adapted to complete automation, and (5) in many situations it may be more economical than other processes for the removal of the use increment of mineral substances.

Objective and Scope of Investigation

The general purpose of this investigation was to ascertain the feasibility of using ion exchange for the removal of the mineral portion of the use increment in sewage prior to re-use. Specifically, the purpose was (1) to optimize the operating variables of a strong acid cation-weak base anion exchange system for the partial demineralization of brackish waste water, (2) to develop a rational basis for the design of partial demineralization systems for municipal use, (3) to identify the limitations and the operational difficulties, and (4) to determine the economic feasibility of the process in comparison with other methods.

The removal of living organisms and trace amounts of other pathogenic substances was not considered and no attempt was made to optimize pre-deionization treatment that served to remove turbidity, organic material, and taste and odor producing substances. The acceptability of the product was based upon the concentration of the common ions and organic material as measured by dichromate chemical oxygen demand (COD) and carbon chloroform extract (CCE), threshold odor, and turbidity.

Previous Work

Although the literature on ion exchange is extensive, there was at the beginning of this investigation little data concerning its application to brackish water or to the production of water for domestic use where appreciable concentrations of dissolved solids are permissible. In 1944, Bachman [3] discussed the economy of demineralizing highly alkaline waters by cation exchange followed by decarbonation, while in 1956, Yoseph [4] described a 3000 gpd demineralization plant for producing potable water from brackish water. In 1952, in an effort to achieve a more economical system, Spiegler, Juda, and Carron [5] reported exchange efficiencies [me (milliequivalents) ions removed from total feed/me new regenerant] of 70 per cent for strong acid resin. They employed the acid regenerant countercurrently and practiced regenerant re-use and an elaborate and strict conservation regimen. Recently, Kunin [6] and Sturin [7] have described processes using three beds of weak electrolyte resins which can be regenerated with high efficiency and produce low solids water from feeds containing up to 3000 mg/ℓ of dissolved minerals. In 1965, Schmidt and Odland [8] reported the SUL-biSUL process to be cheaper than electrodialysis for demineralizing a water containing 1550 mg/ℓ dissolved solids.

In treating sewage or other waters high in turbidity and organic matter, fouling can occur due to irreversible sorption of multivalent ions, build-up of organic slimes, and attachment of particulate matter including bacteria. Strong base resins are the most susceptible to fouling, but according to Kunin [9] they can be protected by sorbing organic ions first on scavenger resins cleansed with brine. Abrams [10] has pointed out the advantages of the weakly basic phenolic resins for reversible sorption of ABS and other organic molecules, while Wilson [11], Frisch and Kunin [12], and Ward and Easterly [13] have discussed the mechanism of fouling of strong base anion resins. Elinsser and his co-workers [14] have reported reasonable success in renovating fouled strong base resins used to reduce nutrients in secondary effluents. Sturla [7] reported 65 per cent removal of organic material from brackish water treated by the Kunin ion exchange process.

LABORATORY INVESTIGATION

The laboratory investigation was divided into two broad phases: (1) optimization of operating variables such as flow rate, regenerant concentration, level of regeneration, etc. and (2) application of the optimized system to the partial removal of dissolved minerals in effluent from an activated sludge plant.

The ion exchange system chosen for evaluation consisted of strong acid (Duolite C-25 or Amberlite 200) and weak base resins (Amberlite IRA-93) in fixed beds with a degasifier between the beds to remove carbon dioxide. The resins were chosen arbitrarily from consideration of the manufacturers' literature and no further attempt was made to find experimentally the most suitable of the many resins available. Storage was

provided following the cation exchanger so that feed for the anion exchanger was uniform in quality.

Phase I. Optimization

Optimization studies were first conducted with feeds consisting of a mineralized well water containing 770 mg/ℓ of total dissolved solids (TDS) or of dilute hydrochloric or sulfuric acids. Finally, a complex synthetic water containing about 2000 mg/ℓ TDS was used to verify the optimized operating conditions at a higher dissolved solids concentration. Extensive studies of regeneration included the effect of: several regenerant chemicals, flow rate and direction, step regeneration with re-use of chemicals, and volume and kind of rinse water. These investigations, which are described in detail elsewhere [15], led to a number of conclusions applicable to the partial demineralization of sewage.

1. A maximum sustained exchange efficiency of 93 per cent was obtained at a regeneration level of 1.25 lb H_2SO_4/cu ft (20 g/liter) by saving and re-using part of the spent acid. Correcting for product water used for rinse and regenerant make up, the maximum sustained operating exchange efficiency was 84 per cent. The regenerant was 5 per cent commercial sulfuric acid applied countercurrently (upflow) at 2 gpm/cu ft (16 bed volumes/hour), a rate sufficient to prevent calcium sulfate precipitation within the bed. Without acid re-use, the maximum sustained exchange efficiency was 80 per cent and the operating exchange efficiency was 70 per cent. The operating exchange capacity was 7.2 K.r/cu ft (330 me/ℓ) with leakage varying from 20 to 25 per cent. For small installations, acid re-use did not appear economically justifiable because of increased equipment and labor costs.

2. The most economical regeneration practice for anion resins was found to be 1 lb ammonia/cu ft (16 g/liter) in 2 per cent solution applied countercurrently (upflow) at a rate of 1 to 2 gpm/cu ft (8 to 16 bed volumes/hour). For resin exhausted with sulfate, efficiencies declined at regeneration rates above 1 gpm/cu ft. The maximum operating exchange efficiency was 86 per cent with an operating exchange capacity of 17.9 Kgr/cu ft (820 mg/l) at an average leakage of about 35 per cent. The nature of the ion leakage could be controlled to some degree by the amount of breakthrough permitted.

3. Adequate mixing for the production of potable water can be obtained with 11 to 16 cm/cu ft (1.3 to 2.1 bed volumes), which is appreciably less than that recommended for industrial practice. It is unnecessary to backwash in every cycle if the feed water is filtered.

Phase II. Demineralization of Sewage

The tertiary treatment plant used for the investigation of sewage recycling consisted of a sand filter and a cation filter (to provide "pretreatment") and cation and anion exchangers. A degasifier for the removal of carbon dioxide was located between the cation and anion units. The filter contained 60 cm of sand with an effective (10 per cent) size of 0.55 mm and a uniformity coefficient (ratio 60:10 per cent size) of 1.38. The cation filter contained 152 cm of Pittsburgh 8-30 mesh (2.38-0.42 mm) SGL activated carbon. Duolite C-25 or Amberlite 200 resins of 16-50 mesh (1.19-0.297 mm) size were used in the cation units with a bed depth of 90 cm. Amberlite IRA-93 resin of 16-50 mesh size was used in the anion unit at a bed depth of 90 cm.

The feed water was obtained from a standard rate activated sludge plant which treated 1.5 gpm of City of Richmond sewage, a weak domestic sewage containing 150 to 200 mg/ℓ BOD and 200 to 500 mg/ℓ COD as determined from 24-hour composite samples. The plant typically removed about 90 per cent of the BOD and 75 per cent of the COD. Normally, the effluent contained a total COD of about 80 mg/ℓ, a turbidity of 30 Jackson candle units (JCU), and a threshold odor of 12 (cf. Table II).

Approximately 600 gallons of secondary effluent were collected for each series of runs. After analysis for the principal ions, several salts were added to increase the TDS to 1200 mg/ℓ and chlorine and mercuric chloride were introduced to prevent decomposition. In two series of tests there was no further treatment other than settling. In other tests the salted effluent was flocculated with 300 mg/ℓ of alum and, after settling, the clarified supernatant liquor was pumped to a storage tank. Feed was applied downflow to the sand and carbon filters at a rate of 2.72 gpm/sq ft (1.77 liters/sec-sq m) and to the cation and anion exchangers at a rate of 3.5 gpm/cu ft (7.8 liters/sec-cu m). The sand filter was generally backwashed after 4 hours of operation. No other units were backwashed after the start of a test series.

Feed to the cation exchanger was stopped when the effluent pH increased to 4.0 while feed to the anion exchanger was halted when the pH of the combined effluent reached 7.0. Occasionally the effluent pH was as low as 6.8 due to difficulties in maintaining strict control over the small flows. In each series the adsorption-regeneration cycles were continued until three consecutive runs gave nearly identical results, thus indicating that cyclic stability had been reached.

Regeneration generally followed the practice established in Phase I. The cation exchanger received 1.25 lb/cu ft of 1.05 normal sulfuric

held at an upflow rate of 2 gpm/sq ft. Filter was put upflow at the same rate with 10.9 gal of demineralized water per sq ft wash. The cation exchanger was regenerated with 0.52 lb/cu ft of 1.0N normal ammonia at an upflow rate of 2 gpm/cu ft and rinsed upflow at the same rate with 13.3 gal/cu ft of resin (1.0 bed volumes).

Results: Ion Exchange Without Pretreatment

Two series of runs were made in which the settled secondary effluent was applied to the ion exchange beds with neither coagulation nor filtration. Ion exchange removed 73 to 79 per cent of the total COD, 85 to 94 per cent of the turbidity, 78 to 83 per cent of the color, 93 to 94 per cent of the odor, and 80 to 95 per cent of the dissolved minerals. However, the COD increased during each run at rates of 0.04 to 0.1 mg/l by 25 to 100 per cent. The ratios of average efficiency was 2:1 for each few Duolite C-25 and 75 per cent for Amberlite 200. The total capacity (efficiency of IRA-93 declined from 75 per cent in the first run to 17 per cent in the tenth with a corresponding increase in turbidity. Comparable to the product obtained with flocculated and filtered secondary effluent, the performance was very poor. The product was unacceptable.

Results: Ion Exchange With Pretreatment

Because the performance of the two cation exchangers, as measured by suitable quantity, color, turbidity and leakage, was similar in both Phase I and Phase II, only Duolite C-25 resin was used in this series. The filtered settled secondary effluent was pretreated by coagulation, sand filtration, and carbon filtration. The sand filter, carbon filter, cation exchanger, and anion exchanger were operated simultaneously in a closed train and the stored and blended effluent from the 12th to the

herein is based on the necessity of both sand and carbon filtration. However, mechanical flocculation and sedimentation have not been included in the analysis because of the likelihood that the coagulated activated sludge effluent can be directly applied to the filter. It is on this basis and with consideration of the laboratory results shown in Table II, that the schematic flow diagram shown in Figure 1 was prepared. The carbon column is designed to operate upflow because exhaustion increases the weight of the carbon particles, causing them to remain at the bottom from whence they can be readily removed for regeneration.

The cost estimates based on Figure 1 are shown in Table III conform to Office of Saline Water (OSW) recommendations [18] with the following exceptions: (1) No charge was made for raw water since it is available at the site. (2) Interest during construction was assumed to be 2 per cent because a twelve-month construction period is adequate for ion exchange plants. (3) Maintenance, labor and supplies costs were doubled. (4) Operating labor was added because of its uncertainty. Costs, which were obtained from OSW [16], Perry and Devid [19], Manns [20], Page [21], and local companies, were corrected to an EMR Construction Cost Index [22] of 1970 which corresponds to late 1965 prices. The freight distances assumed were: 1000 miles (1600 km) for fertilizer, 2000 miles (3200 km) for carbon and polyelectrolyte, and 500 miles (450 km) for other chemicals. Since the 1 mgd (3,785 cu m/day) ion exchange plant is well below optimum in size, the "six-tenths cost factor" [23] was used to compute the cost of facilities for a 10 mgd (37,850 cu m/day) plant. Using the method proposed by Higgins [24] for the thermal recovery of nitric acid and ammonia and including amortization, maintenance, and 10 per cent make-up chemicals, it appears that the cost of ion exchange in a

10 mgd plant can be reduced to $0.10/1000 gal (3.79 cu m) exclusive of operating labor.

The pretreatment (coagulation and sand and carbon filtration) portion of the plant shown in Figure 1 is similar to the 2.5 mgd (9,500 cu m/day) tertiary sewage treatment plant at Lake Tahoe which Roderick and Culp [17] reported cost $625,000 (including engineering) to construct and $0.144/1000 gal to operate, including labor and amortization over a life of 20 years at 3 per cent interest. The operating costs of a 10 mgd plant were estimated to be $0.108/1000 gal. However, on the basis used for estimating the ion exchange costs herein, the estimated cost of pretreatment would be $0.19/1000 gal in a 1 mgd plant and $0.15/1000 gal in a 10 mgd plant excluding operating labor. A proposed plan to recover alum might reduce these costs by $0.03/1000 gal.

Operating labor costs are speculative. Automatic ion exchange plants operate with only about two hours of attention per day. The pretreatment plant would probably require more attention. However, direct recycling might require continuous surveillance by competent personnel which with labor overhead would cost as much as $0.14/1000 gal in a 1 mgd plant and $0.014/1000 gal in a 10 mgd plant. To this must be added charges for chemical and biological analyses.

Chlorination would add about $0.006/1000 gal in a 10 mgd plant to $0.01/1000 gal in a 1 mgd plant. Disposal of waste by injection at a depth of 7000 ft (2100 m) might, according to Koenig [25], cost about $0.005/1000 gal for a 10 mgd plant to $0.02/1000 gal of product for a 1 mgd plant.

Total costs, including amortization, labor, chlorination, and waste disposal, are estimated to be about $0.52/1000 gal in a 1 mgd plant and about $0.23/1000 gal in a 10 mgd plant.

Comparison With Other Methods

Higgins [24] has estimated a cost of $0.069/1000 gal in a 10 mgd moving-bed ion exchange plant. Adding amortization for a life of 15 years and using West Coast (USA) prices for make-up acid, the cost would be about $0.12/1000 gal exclusive of operating labor, pretreatment, and chlorination. This is comparable to the $0.10/1000 gal, estimated for a 10 mgd plant of the fixed-bed type (Figure 1).

Smith and Eisenmann [26] estimated the cost of removing 350 mg/ℓ of solids from 10 mgd of secondary effluent to be $0.092/1000 gal by electrodialysis exclusive of labor and assuming amortization over 25 years. However, on a comparable basis of estimation, ion exchange would cost about 10 per cent less than electrodialysis if acid and ammonia could be recovered by heat as proposed by Higgins [24]. Pretreatment, labor, and chlorination costs would be the same as those for ion exchange.

With the data given by Neal [27], it can be shown that the removal of 350 mg/ℓ from 10 mgd by split-treatment distillation would cost about $0.20/1000 gal including operating labor. The cost of pretreatment, chlorination, and waste disposal would be nearly the same as for ion exchange.

CONCLUSIONS

1. Provided the thermal recovery of acid and base can be accomplished at the costs herein estimated, fixed-bed ion exchange appears to be economically superior to electrodialysis, to moving-bed ion exchange, and to distillation.
2. If thermal recovery of chemicals is not practiced, ion exchange appears to be comparable in price with electrodialysis only if chemicals are produced in the vicinity of the ion exchange plant.

3. Although the quality of the process stream is (or can readily be made) satisfactory, split-treatment blending cannot be expected to provide suitably low levels of nitrogen regardless of the process used.

4. Pretreatment of activated sludge effluents is necessary for ion exchange, electrodialysis, and distillation and it costs as much as the processes for the reduction of dissolved solids. The use of sewage spreading or other recharge methods which reduce nitrogen, BOD, phosphate, and turbidity may offer an economic advantage for partial pretreatment as well as serving for storage to equalize the load on the ion exchange system.

5. The waste stream of an ion exchange plant is only 3.3 per cent of the blended product stream. In many situations it may be discharged without special treatment provisions.

6. A use increment of 500 mg/l can be removed from 10 mgd (37,850 cu m/day) of secondary effluent by ion exchange at an estimated cost of less than $0.25/1000 gal ($.19 cu m) including pretreatment, operating labor, amortization, and disposal of wastes providing the regenerant can be recovered at the costs estimated herein.

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TABLE I

REPRESENTATIVE USE INCREMENTS FOR 22 U.S. CITIES [27]

Constituent	Use Increment, mg/ℓ		
	Min.	Max.	Avg.
Sodium	8	101	66
Calcium	1	50	18
Magnesium	0	15	6
Ammonium	0	36	15
Chloride	6	200	74
Sulfate	12	57	28
Bicarbonate	-44	265	100
Nitrate	-5	26	10
Silica (SiO_3)	9	22	15
Phosphate (PO_4)	7	50	24
TDS	128	541	320
BOD	8	27	16
COD	22	159	87

TABLE II

RESULTS: ION EXCHANGE WITH PRETREATMENT

Constituent	Concentration, mg/ℓ				
	Activated Sludge Effluent[a]	Following Coagulation and Sedimentation	Following Sand Filtration	Following Carbon Filtration	Following Ion Exchange
Sodium	-	147	-	148	53
Potassium	-	12	-	13	7
Calcium	-	97	-	97	1.8
Magnesium	-	69	-	69	0.2
Ammonium	-	3	-	3	3
Sum Cations	-	328	-	330	67
Chloride	-	108	-	108	117
Sulfate	-	272	-	270	10
Bicarbonate	-	236	-	254	12
Nitrate	-	113	-	132	29
Nitrite	-	2	-	0	0
Phosphate (PO_4)	-	5	-	3	2
Sum Anions	-	804	-	833	170
Dissolved Solids					
Calculated	-	1172	-	1163	266
Total, 105°C	-	1132	1177	1096	272
Fixed, 600°C	-	856	854	846	212
COD, suspended	39	2	1	1	2
COD, dissolved	43	30	31	8	9
Color, APHA units	20	10	12	0	1
Turbidity, JCU	40	1.5	0.6	0.4	1.3
Threshold odor	13	8	8	2	2

[a]Salts were added to the activated sludge effluent and the mixture was coagulated with approximately 140 mg/liter alum.

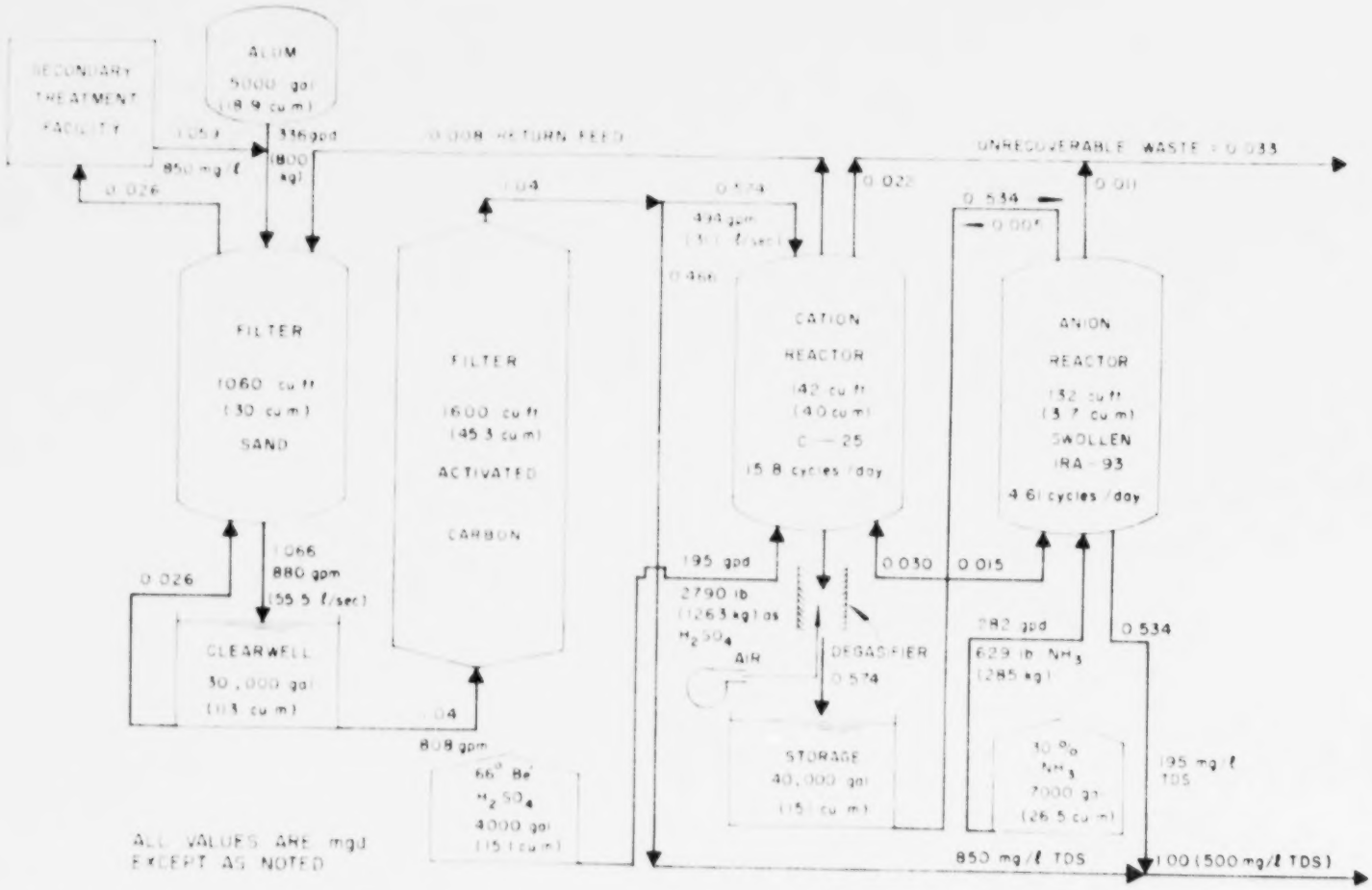

FIGURE 1 SCHEMATIC FLOW DIAGRAM FOR 1.00 mgd (3785 cu m) ION EXCHANGE PLANT TO TREAT 850 mg/ℓ TDS SECONDARY EFFLUENT

TABLE III

ESTIMATED COSTS TO REMOVE 350 mg/ℓ USE INCREMENT BY ION EXCHANGE

Item	Cost, $	
	1 mgd[a]	10 mgd
Capital cost, installation and erection		
Cation reactor, rubber-lined plus resin	23,514	
Degasifier, wood tower and wood basin	17,666	
Anion reactor, rubber-lined plus resin	31,203	
Instruments, fully automatic	18,300	
Acid storage, steel	3,560	
Ammonia storage, steel	4,180	
Pumps and mixers	19,435	
Motors, control panels, and starters	2,340	
Total essential plant cost (TEPC), subtotal	123,393	586,100[b]
Other plant costs		
Product storage @ $14.70/1000 gal	14,700	147,000
Contingencies @ 10 per cent of above	14,810	73,510
Engineering @ 10 per cent of above	16,291	80,861
Interest during construction @ 2 per cent of above	3,684	17,800
Plant site @ $4.42/1000 gal	4,420	44,200
Total plant investment (TPI)	187,203	951,471
Operating expenses for ion exchange		
Amortization. Average life = 12 yrs.,	$/day	58.99	322.90
Electric power @ 1¢/kw hr,	$/day	5.50	55.00
Sulfuric acid @ $41.35/ton delivered,	$/day	58.50	585.00
Ammonia @ $118.70/ton delivered,	$/day	37.40	374.00
Resin addition @ 5%/yr.,	$/day	1.56	12.90
Supplies and maintenance @ 3.0% TPI,	$/day	5.62	28.55
Maintenance labor @ 3.0% TPI + 50% overhead,	$/day	8.43	42.82
Taxes and insurance @ 6.0% TPI,	$/day	11.24	57.10
Interest on working capital @ 3.0% above,	$/day	1.36	10.70
Total cost excluding operating labor,	$/day	188.40	1,489.97
Total cost excluding operating labor,	$/1000 gal	0.19	0.15

[a] See Figure 1.

[b] TEPC except resins multiplied by $(10/1)^{0.6}$, the "six-tenths cost factor" [25].

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